Rheology is the branch of science dealing with the flow and deformation of materials. Rheological instrumentation and rheological measurements have become essential tools in the analytical laboratory for characterizing component materials and final products, monitoring process conditions, as well as predicting product performance and consumer acceptance. Today, rheological instrumentation and rheometry are accepted techniques to more fully characterize, understand, and provide control for crude oil production and transportation. The way in which a particular chemical structure is studied or analyzed, the techniques and instrumentation involved, and how these may be used or modified to solve a problem are paramount.

One of the more important but least utilized aspects of rheology is normal force. It is well known that when a simple liquid such as water is stirred, inertia causes the sample to be displaced away from the axis of rotation. However, when an elastic solution such as polyisobutylene is stirred, it gravitates toward the axis of rotation and creates an inverted vortex. This phenomenon is called rod-climbing or the Weissenberg effect (Figure 1). The effect is due to the generation of normal stress, which acts toward the center of rotation. Normal force is a very sensitive and accurate measurement of the elasticity of a material, and can generate useful information relating the internal structure to the material’s flow behavior. Although there has been much interest in this area, measurement of the elasticity of a material, and can generate useful information relating the internal structure to the material’s flow behavior. Although there has been much interest in this area, measure-ments are encumbered by instrument limitations, especially for lower-viscosity samples. Specifically, the ability to measure small axial force signals at high shear rates is required, since many lower-viscosity fluids do not generate appreciable axial force until the shear rate exceeds 1000 s⁻¹.

This paper details the Differential Pressure Normal Force (DPNF) sensor (ATS RheoSystems/RELOGICA Instruments, Bordentown, NJ), which is based on the principle of differential pressure. Data are shown indicating the sensitivity and precision level that can be achieved using the sensor with a STRESSTECH HR rheometer (ATS RheoSystems/RELOGICA Instruments). Results on an NIST (Gaithersburg, MD) solution are compared to NIST-published results. Also, data are given on a high-crude oil with and without the addition of a small amount of high-molecular-weight polymer. The polymer, added in the ppm range, is used to reduce the pressure drop when pumping the crude oil through a pipeline. This effect is called drag reduc-tion, and the polymer additives employed for this purpose are commonly called drag-reducing agents (DRAs). Since a higher-pressure drop results in more energy consumption for the same amount of work, great care is taken to determine the exact cause of this effect, and to develop a simple, reliable way to measure, control, and maximize it.

**Concept of normal stress: Overview**

The effect of elasticity of a material is attributed to the normal stresses generated during the shear flow. A simple shear flow profile is shown in Figure 2. The flow is in the x direction, while the velocity gradient exists in the y direction. The first normal stress difference, \( N_1 \), is given as \( \tau_{xy} - \tau_{yx} \), while the second normal stress difference, \( N_2 \), is given as \( \tau_{xx} - \tau_{yy} \).

For Newtonian materials such as pure water, both \( N_1 \) and \( N_2 \) are zero in shearing flow. For non-Newtonian materials, \( N_1 \) is positive and much larger than \( N_2 \). \( N_2 \) is normally very small in magnitude and has been reported to be negative for some materials. This suggests that elastic fluids generate extra stress above the shear stress in the direction of the flow. This extra stress in the elastic fluids is generated due to stretching and alignment of the polymer chains along the streamlines. In other words, polymer chains act like a rubber band, and seek to regain the equilibrium configuration, which generates extra tension along the streamlines.

Using different geometries, \( N_1 \) and \( N_2 \) can be measured. For cone and plate geometry, the working equation for \( N_1 \) based on the axial thrust is:

\[
F = \frac{4 R^2}{h} N_1
\]

Where \( R \) is the radius of the plate, and \( F \) is the total axial force on the plates.

This axial force acts to separate the cone and plate. The axial force is what gives rise to the Weissenberg effect described above. In the rheometer, since the plates are held in a fixed position, a transducer mounted to one of the plates can make a direct measurement of this force.

### Figures

**Figure 1** The Weissenberg effect shown by a solution of polyisobutylene (Upolushene® BZ200, BASF Corp., Florham Park, NJ) in polybutane.

**Figure 2** Simple shear flow.

Hence, using Eq. (2), one can determine \( N_1 \) by measuring the axial force in Newtons.

Similar mathematical analysis for parallel plate geometry reveals the following working relationship:

\[
F = \frac{6 R^2}{t} (N_1 - N_2)
\]

Thus, by using cone and plate and parallel plate geometries, with Eqs. (2) and (3) (see below), the individual magnitudes of \( N_1 \) and \( N_2 \) can be determined. However, as mentioned above, \( N_2 \) is very small and/or negative, and therefore generally neglected for practical purposes.

**DPNF sensor**

As the name suggests, the DPNF sensor is based on the principle of measuring the pressure in the air bearing supporting the drive shaft in a rotational rheometer. The air bearing imparts a restoring force equal in magnitude and opposite in direction as a result of the axial force generated by the elastic liquid being sheared between the plates in the rheometer. The DPNF assembly schematic is shown in Figure 3. Upon generation of axial force on the shaft, gas pressure builds up between the two opposing faces of the rotor. A differential between the gas pressure on the opposing faces of the rotor is established, and the axial force acting on the shaft is determined as a product of a constant and the pressure differential between the gas pressures on the two faces.

As shown in Figure 3a, a shaft that holds the different measurement systems (10) rotates in an air bearing. The air bearing holds a body (15) consisting of two ring-shaped sections that surround portions of the shaft (10). Hence, there is an air column forma-tion (11) between the shaft (10) and the body (15). The rotor (12) moves the shaft (10) radially. The axially opposing sides or faces (16 and 17) have the same diameter as the body (15). The body (15) is surrounded by an outer casing (21) through which air returns via a channel (13). The pressure measure-ment device is a differential pressure sensor (18), which is connected to the outlets (19 and 20). The air is conducted to the chamber body (15) via channels (28). Here, F is the restoring force acting on the measurement body shown in Figure 3b.

In Figure 3b, the shaft (10) is shown along with the conventional parallel plate measurement geometry (22). The first measuring body (22) is connected.
to the second fixed measuring body (23) via sample media (24). The shaft on the other end is connected in a conventional way to a drive motor (37). Using this sensor, the authors performed the studies described below.

### Experimental

Rheological measurements were made using a fully automated STRESSTECH HR rheometer equipped with the DPNF sensor (Figure 4). The rheometer is designed with industry-leading torque sensitivity, position resolution, and normal-force capability. As described above, measurement of the elasticity of a material plays an important role in predicting the end-use performance of the material. Therefore, to quantify the difference in the end-use performance, an accurate and sensitive yet robust means of measuring the elasticity of the materials is required.

### Results and discussion

As described above, the study was divided into two parts: 1) DPNF sensor precision and accuracy performance, and 2) normal-force measurements on low-viscosity liquids and application to drag reduction.

1. **DPNF sensor sensitivity study.** As mentioned above, a study on NIST-traceable material was conducted to evaluate the performance of the DPNF sensor. The solution used for the study was Standard Reference Material 2490 (SRM 2490). SRM 2490 consists of a polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane. The solution contains a mass fraction of 0.114 polyiso-

butylene. The mass average relative molecular mass of the polyisobutylene is reported as 1,000,000. The data used were collected from 144 laboratories over a three-year period.

For the normal-force study, steady shear tests were performed from 0.05 to 100 s⁻¹ at 25 °C. A 48-mm/2° cone and plate fixture was used. SRM 2490 was loaded with constant 5.0 N normal force to ensure the exact sample loading history. The sample was trimmed at a 50-µm gap above the target gap to reduce the error in data collection due to edge effects. Rheological data were collected as a function of shear rate. Figure 5 shows the results for this study along with the NIST-published data. As expected, in the terminal zone, N₁ increased with shear rate at a slope of 2 on a log/log plot. The following fundamental equation can be used to fit the relationship in the terminal zone:

\[
N_1 = A \frac{\gamma^m}{\gamma^m}
\]

Where A and m are constants, with m being typically in the range of 1 < m < 2. As the shear rate decreases, the value of m approaches 2.

Using Eq. (3), for the NIST sample, the fit for shear rate is in the 0.05–1.0 s⁻¹ range, giving the following relationship:

\[
N_1 = 58.3 \frac{\gamma^2}{\gamma^2}
\]

As the results indicate, the STRESSTECH HR rheometer data and NIST-published data agree well at intermediate to high shear rates, where the measured axial force is above 0.06 N.

The DPNF sensor equipped with the STRESSTECH HR agrees with the published results to the lower limit provided by NIST of 0.2 s⁻¹ at an axial force of 0.005 N. Note the ability of the DPNF sensor to provide accurate N₁ resulting from 0.1 s⁻¹ at an axial force of 0.00055 N. These results are below those published by NIST for SRM 2490.

As mentioned above, normal force is a result of elasticity in a material. Other rheological properties also manifest themselves as a result of elasticity. Viscosity is another method for quantifying the elastic behavior of materials. Since all of these measurements are in effect based on the same material property, they can be mathematically related to each other. Hence, the normal force generated during steady shear experiments can be compared to viscoelastic properties such as dynamic viscosity (η′), dynamic rigidity or storage modulus (G′). In the terminal zone, using fundamental relationships, it can be shown that:

\[
G' (\omega) = \frac{N_1 (\gamma)}{2 \pi \gamma^2}
\]

From Eq. (6), G′ = N₁. To confirm this relationship, oscillatory experiments on SRM 2490 were performed from 0.001 to 100 rad/sec in the linear viscoelastic region using the same geometry at 25 °C to generate viscoelastic data. The results are plotted in terms of G′, G″, and η* versus frequency (Hz) in Figure 6. Using Eq. (6), the data collected in the steady shear and dynamic study are compared in Figure 7. As can be seen, one approaches the terminal zone, the data agree, as expected.

2. **Normal-force application in drag reduction.** In the simplest terms, drag defines the power or energy required to maintain the given output flow rate. The drag coefficient or friction factor can be measured as the ratio of work done by force divided by kinetic energy carried by the fluid. In the laminar flow process, the drag coefficient is inversely proportional to the Reynolds number, while in the turbulent process, the drag coefficient is independent of the Reynolds number and much higher than in laminar flow. As the drag increases, a higher pressure drop or higher pumping capacity is required to maintain the same flow rate.

In 1948, British chemist B.A. Toms discovered that by adding a small amount (ppm) of high-molecular-weight polymer to water, a nearly 80% reduction in drag could be achieved. This study triggered new interest in the use of rheological measurements to understand this effect and, as a result, much work has been done to comprehend the exact mechanism behind this flow behavior. Theory attributes this phenomena to change in boundary layer formation. Due to the presence of long-chain polymers, the boundary layer thickness increases and therefore acts as a smoothing layer, and hence pressure drop is reduced. Thus far, none of the experimental or theoretical studies has shown a clear relationship between turbulence and polymer interaction. However, due to the presence of DRAs, the elasticity of the fluid changes drastically, which can be measured by changes in normal force.

Experiments on a crude oil with and without DRA have been performed in an effort to find a simple, fast, and reliable way to characterize the effect and performance of the DRA. Each crude oil sample, along with a Newtonian standard, was run in a couette 25-mm measuring system to determine the effect of the DRA polymer on the shear viscosity (Figure 8). As documented in the literature, the sharp increase in viscosity is due to the onset of secondary flow called Taylor vortices. In the literature, it has been shown that the shear rate at which this effect manifests itself trends with viscosity; however, it is interesting to note that while the crude oil sample with DRA has a higher viscosity, it...
transitions into secondary flow at lower shear rates. This may be attributed to an extensional or elastic effect in the lower section of the measuring system. Thus, an elasticity measurement may allow quantification of the DRA mechanism.

To investigate this phenomenon, and in an attempt to determine the contribution of extensional viscosity and elasticity, $N_1$ was measured for the same three samples with parallel plate geometry, since the parallel plate geometry does not suffer from the same secondary flow limitation as the measuring system used. $N_1–N_2$ and viscosity were measured in a shear rate sweep using a 25-mm parallel plate geometry at a 30-μm gap run at 25 °C. The results for the three samples are plotted in Figure 9. Included are data on the same crude DRA sample run three successive times.

The crude oil with DRA generated significant normal force upon shearing, while the Newtonian standard and neat crude oil did not generate any normal force, and in fact showed a decrease. The decrease in $N_1–N_2$ for the two inelastic samples is due to sample inertia causing the sample to move away from the axis of rotation. In addition, it was also found that the elasticity for the crude DRA was reduced because of shearing. This suggests that breakage or degradation of the polymer chains may be taking place as a result of high shear rates. This breakdown may also be occurring in the pipeline flow in which the DRAs are being used.

Since crude oil is pumped and processed in the field at elevated temperatures around 100 °C, DRA performance must be studied at these conditions. However, due to the volatile nature of the sample containing light hydrocarbons, a measuring system is needed that will provide a head pressure of up to 60 psig to maintain the sample integrity while allowing normal-force measurements. To accomplish this, a patented Sealed Cell accessory with parallel plate geometry was used (ATS RheoSystems/REOLOGICA Instruments) (Figure 10). The Sealed Cell is the only commercially available system to allow normal-force measurements under pressure, uses a proprietary design to pressure the sample while not mechanically contacting the rotating shaft, and thus allowing the axial force to be measured.

The two crude oil samples were tested at 93 °C at a head pressure of 60 psig. A shear rate sweep from 1000 to 50,000 1/sec was performed with a 25-mm parallel plate with a run gap of 50 μm. The axial force generated was collected as a function of shear rate. $N_1–N_2$ is plotted versus shear rate in Figure 11. As shown previously, at room temperature, the DRA crude demonstrated strong elasticity, while the neat crude did not exhibit any appreciable elasticity.

For this sample, using Eq. (3), the following empirical relationship can be established between shear rates 2000 and 20,000 1/sec:

$$N_1 = 0.0017 \gamma^1.33$$

(6)

The addition of DRA to crude oil causes a slight increase in the viscosity and a significant increase in the elasticity of the sample. As the DRA reduces the pressure drop, the elasticity, not the viscosity, appears to be the controlling resistance in terms of drag reduction. This elasticity can be quantified by making reliable normal-force measurements under field conditions.

**Conclusion**

The studies presented here demonstrate both the sensitivity and performance of the DPNF sensor for reliable rheological measurements, as well as the ability of these measurements to help solve real-world problems. Using DRAs, one can alter the elasticity of the fluid and hence its flow properties. Application of DRAs has been a very economical approach to reducing the pressure drop across the flow lines and therefore lowering pumping costs. The present study shows how different fluids can be easily analyzed to demonstrate the effect of DRAs on the elasticity of the fluid. The rheological measurements have been a fast, accurate, and economical way to determine flow properties.

Most researchers and manufacturers count on rheological measurements to develop products with a competitive edge in the marketplace. A reliable research-level rheometer and a thorough understanding of rheological measurements are now a necessity for success in today’s market.

**References**


**Additional reading**


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**Figure 7** Relationship between normal-force and viscoelastic properties of NIST 2490.

**Figure 8** Effect of DRA on shear viscosity in couette 25-mm measuring system at 25 °C.

**Figure 9** Effect of elasticity on crude oil using DRA using parallel plate system at 25 °C.

**Figure 10** Sealed Cell with parallel plate geometry.

**Figure 11** Normal stress/shear rate relationship for crude oil with and without DRA in Sealed Cell.


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